

Iodide, colorimetric, ceric-arsenious oxidation, automated-segmented flow

Parameter and Code:

Iodide, dissolved, I-2371-85 (mg/L as I): 71865

1. Application

This method may be used to determine iodide in fresh water and brines containing from 0.001 to 0.060 mg/L of iodide. Greater concentrations need to be reduced by dilution; however, samples containing concentrations greater than 1 mg/L need to be analyzed by the bromine oxidation method (method I-1370).

Absorption cell ----- 15 mm

Wavelength ----- 410 nm

Cam ----- 30/h (2/1)

2. Summary of method

The method is based on the catalytic effect of iodide on the ceric-arsenious oxidation reaction in acid solution. In the presence of a small amount of iodide, the reaction follows first-order reaction-rate kinetics-and at a given temperature and for a given reaction time, the extent of reduction of ceric ion is directly proportional to the iodide concentration. Photometric measurement of the absorbance of the solution permits evaluation of the extent of the reaction (Mitchell, 1966).

5. Reagents

5.1 *Arsenious acid solution*, 2.782 g/L: Add 2.782 g primary standard As_2O_3 to approx 500 mL demineralized water in a 1,000-mL volumetric flask. Slowly add 3 mL concentrated H_2SO_4 (sp gr 1.84). Warm the mixture until the As_2O_3 is dissolved. Cool and dilute to 1,000 mL with demineralized water. Add a small piece of metallic arsenic to stabilize the solution.

5.2 *Ceric sulfate solution*, 1.584 g/L: Add 600 mL 2.5M H_2SO_4 and 1.584 g anhydrous $\text{Ce}(\text{HSO}_4)_4$ to a 1,000-mL volumetric flask. Warm the mixture and stir occasionally until a clear solution is obtained. Cool and dilute to volume with 2.5M H_2SO_4 .

5.3 *Iodide standard solution I*, 1.00 mL = 0.100 mg I^- : Dissolve 0.1308 g KI crystals, dried overnight in a sulfuric acid desiccator, in demineralized water and dilute to 1,000 mL.

5.4 *Iodide standard solution II*, 1.00 mL = 0.010 mg I^- : Dilute 100 mL iodide standard solution I to 1,000 mL with demineralized water.

5.5 *Iodide standard solution III*, 1.00 mL = 0.0005 mg I^- : Dilute 50.0 mL iodide standard solution II to 1,000 mL with demineralized water.

5.6 *Iodide working standards*: Prepare a blank and 500 mL each of a series of iodide working standards by appropriate quantitative dilution of the iodide standard solution III as follows:

3. Interferences

3.1 Most substances normally present in natural water do not interfere.

3.2 Bromide ion does not interfere.

3.3 Low iodide values will be obtained if the pH of the water samples is less than 3 or if the iron concentration exceeds 500 $\mu\text{g/L}$.

4. Apparatus

4.1 *Technicon AutoAnalyzer II*, consisting of a sampler, proportioning pump, cartridge manifold, colorimeter, voltage stabilizer, recorder, printer, and heating bath.

4.2 With this equipment the following operating conditions have been found satisfactory for the range from 0.001 to 0.060 mg/L:

Iodide standard solution III (ml)	Iodide concentration (mg/L)
0.0	0.000
2.0	0.002
5.0	0.005
10.0	0.010
20.0	0.020
40.0	0.040
60.0	0.060

5.6 *Sulfuric acid, 2.5M*: Cautiously add 139 mL concentrated H_2SO_4 (sp gr 1.84) to demineralized water, cool, and dilute to 1 L.

6. Procedure

6.1 Set up manifold (fig. 27).

6.2 Allow colorimeter and recorder to warm up for at least 30 min.

6.3 Adjust the baseline to read zero scale division on the recorder with all reagents, but with demineralized water in the sample tube.

6.4 Place a complete set of standards and a blank in the first positions of the first sample tray, beginning with the most concentrated standard. Place individual standards of differing concentrations in approximately every eighth position of the remainder of this and subsequent sample trays. Fill remainder of each tray with samples.

6.5 Begin analysis. When the peak from the most concentrated working standard appears

on the recorder, adjust the STD CAL control until the flat portion of the peak reads full scale.

7. Calculations

7.1 Prepare an analytical curve by plotting the height of each peak versus its respective iodide concentration.

7.2 Compute the iodide concentration of each sample by comparing its peak height to the analytical curve. Any baseline drift that may occur must be taken into account when computing the height of a sample or standard peak.

8. Report

Report iodide, dissolved (71865), concentrations as follows: less than 0.1 mg/L, three decimals; 0.1 mg/L and above, two significant figures.

9. Precision

Precision data are not available for this method.

Reference

Mitchell, C. G., 1966, Semimicroanalytical method for the determination of iodide in water: U.S. Geological Survey Water-Supply Paper 1822, p. 77-83.

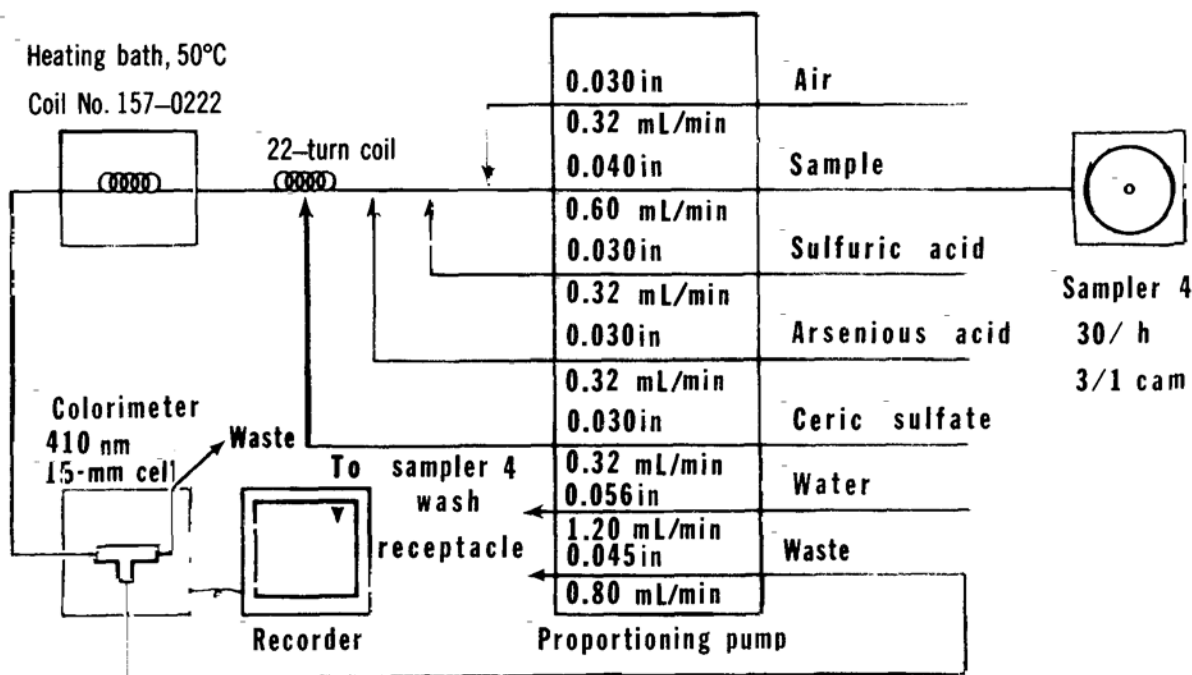


Figure 27.—Iodide, ceric-arsenious oxidation manifold